I. Temperature Scales, Uncertainty, and Traceability

II. Thermocouples and Thermocouple Wires

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Outline

Temperature Scales

Overview of thermocouples (TCs) and reference functions

Traceability

Thermocouple construction types

Laws of thermocouple circuits

Electrical characteristics, differential thermocouples, extension wires, and feedthroughs

Limitations on thermocouple performance

Calibration uncertainties and methods

Calibration of used thermocouples

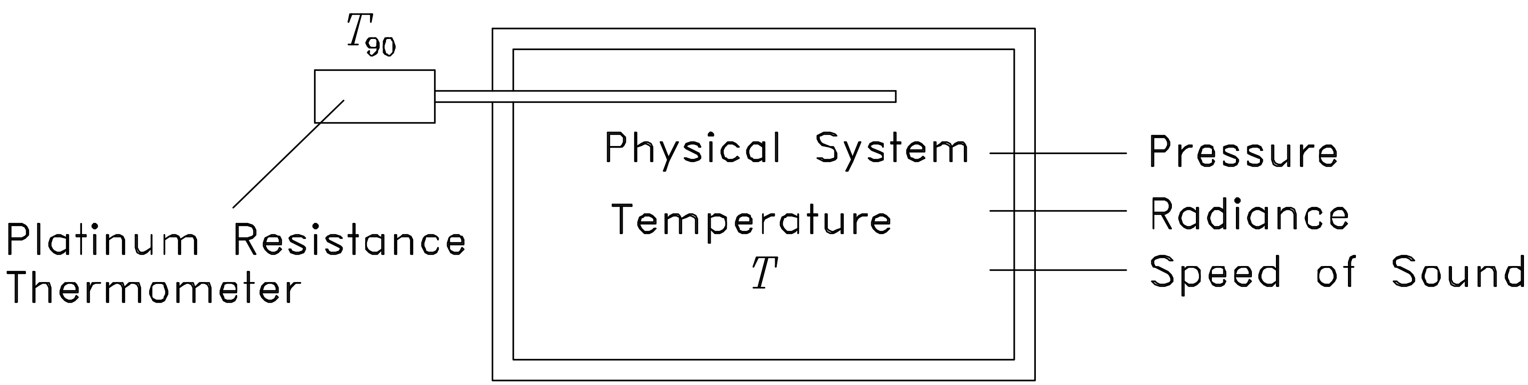
Resources

What is Thermodynamic Temperature?

Thermodynamic temperature (*T*) satisfies all of the laws of thermodynamics

Primary Thermometer is an apparatus whose temperature can be calculated from other measured properties (pressure, radiance, speed of sound, etc.).

Examples of Physical Systems: Black body radiation
Speed of sound in ideal gas



Typically, $T - T_{90}$ is measured, where T_{90} = temperature on the International Temperature Scale of 1990 (ITS-90)

What is the International Temperature Scale of 1990 (ITS-90)?

The ITS-90 is an approximation of the thermodynamic temperature scale.

The ITS-90 is a specific recipe for "realizing" temperature, including:

- Defined thermometer types
- Reproducible fixed points (triple point of water, freezing point of silver, etc.)
- The triple point of water is defined as 273.16 K (0 °C = 273.15 K)

Defining instruments relevant to semiconductor thermometry:

- Standard Platinum Resistance Thermometer (SPRT) from 13.6 K to 962 °C
- Spectral Radiation Thermometer from 962 °C to all higher temperatures

The ITS-90 replaces earlier International Temperature Scales:

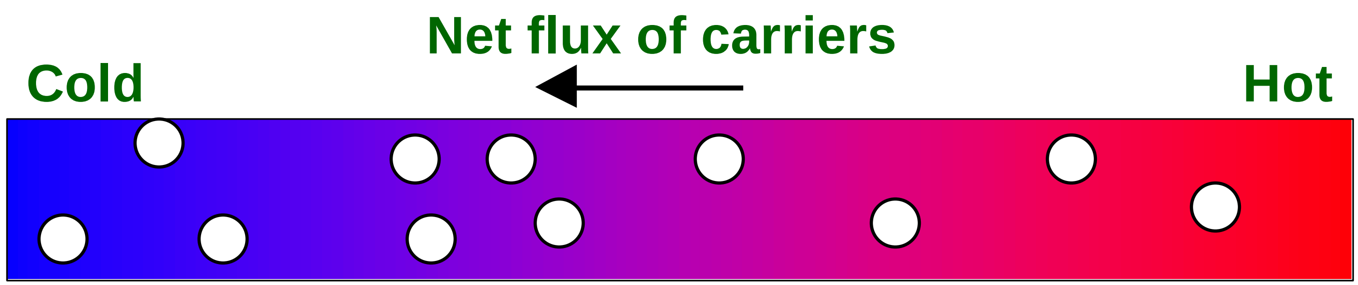
1968	(IPTS-68)
1948	(ITS-48)
1927	(ITS-27)

The IPTS-68 is still in use by some industries

Why use the International Temperature Scale of 1990 (ITS-90)?

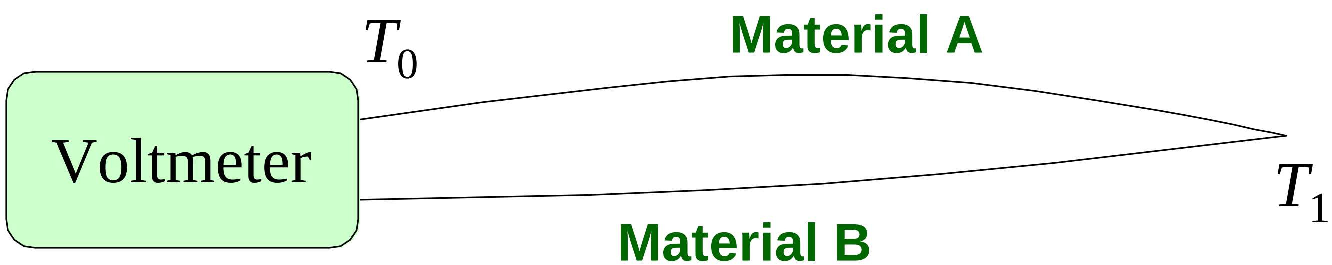
- The ITS-90 is much easier to realize than true thermodynamic temperature.
- The ITS-90 is internationally accepted.
- For all present commercial applications, the thermodynamic inconsistency of the ITS-90 is negligible.
- Establishment of temperature uncertainty more straightforward on the ITS-90 than with “Process measurement scales”.
- Consistency of temperature readings worldwide and across applications.

What is a Thermocouple?



Hotter carriers travel farther before equilibrating with the crystal lattice than cold carriers.

Consequence: charge imbalance when crystal is in thermal gradient.



Net electromotive force = emf = $E = E_A(T_1, T_0) - E_B(T_1, T_0)$

Advantages of Thermocouples

- Cheap
- Wide temperature range (–270 °C to 2100 °C)
- Small (down to 0.25 mm diameter)
- Easy to integrate into automated data systems

Disadvantages of Thermocouples

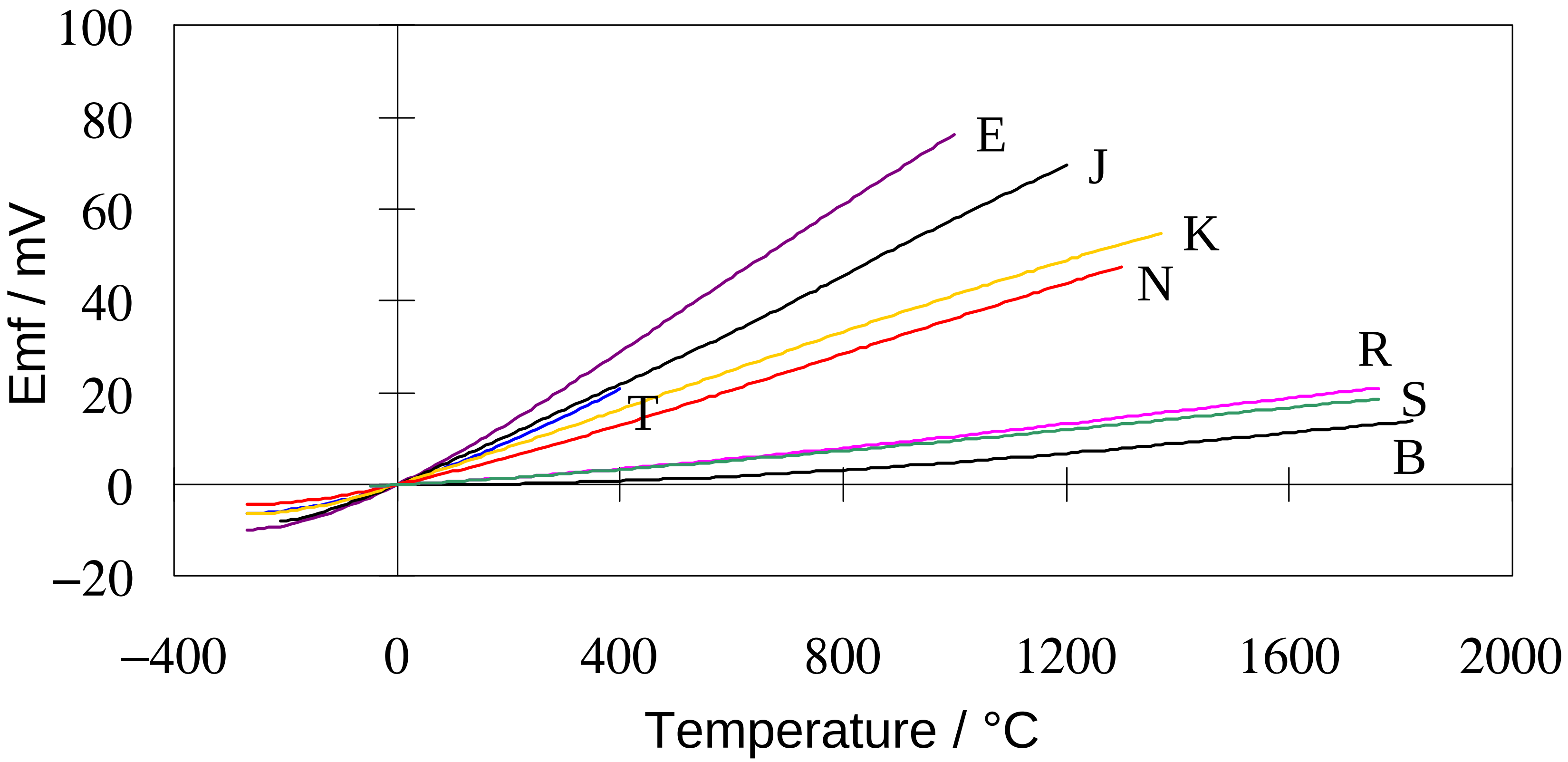
- Small signals, limited temperature resolution (1 mK to 1 K)
- Thermocouple wires must extend from the measurement point to the readout. Signal generated wherever wires pass through a thermal gradient.
- At higher temperatures, thermocouples may undergo chemical and physical changes, leading to loss of calibration.
- Recalibration of certain types of thermocouples or in certain applications is very difficult.

Letter-designated Thermocouple Types

TC type	Ref. function range, °C	Nominal composition, majority component in italics: Positive leg	Negative leg
B	0 to 1820	*platinum*-30% rhodium	*platinum*-6% rhodium
E	–270 to 1000	*nickel*-chromium alloy	*copper*-nickel alloy
J	–210 to 1200	iron	*copper*-nickel alloy
K	–270 to 1372	*nickel*-chromium alloy	*nickel*-aluminum alloy
N	–270 to 1300	*nickel*-chromium-silicon	*nickel*-silicon-magnesium
R	–50 to 1768	*platinum*-13% rhodium	platinum
S	–50 to 1768	*platinum*-10% rhodium	platinum
T	–270 to 400	copper	*copper*-nickel alloy

The letter designations define emf versus temperature only — not composition

Emf-Temperature Relationships for the 8 Letter-Designated Thermocouple Types



Notation: *E* = Emf = Electromotive Force = Thermoelectric Voltage
S = *dE*/*dT* = Seebeck Coefficient = Sensitivity

Thermocouple Reference Functions

Sources:

- Reference functions for letter-designated TC types in ASTM E230, IEC 584, NIST Monograph 175
  (ASTM = American Society for Testing and Materials,
  IEC = International Electrotechnical Commission)
- Reference functions for non-letter designated types in: ASTM E1751, E988

Cautions:

- Only a set of spools is guaranteed to match reference function
- Some reference functions are not smooth near 0 °C

Manufacturer develops alloy

↓

Emf versus temperature measured. Reference function fit to data.

↓

Alloy manufactured to match reference function.

Choosing a Thermocouple Type

type E: High Seebeck coefficient, homogeneous materials. Good for low temperatures.

type J: Cheap!

type K: Fairly cheap high temperature thermocouple.

type N: Best base metal thermocouple for high temperatures.

type T: Homogeneous materials. Direct connection of differential pairs to voltmeters.

Use type K, E, or T at room temp., type K up to 200 °C, type N in the range 300 °C to 600°C, type N or K above 600 °C

type R, S: Noble metal thermocouple for range 0 °C to1400 °C.

type B: Noble metal thermocouple used from 800 °C to 1700 °C.

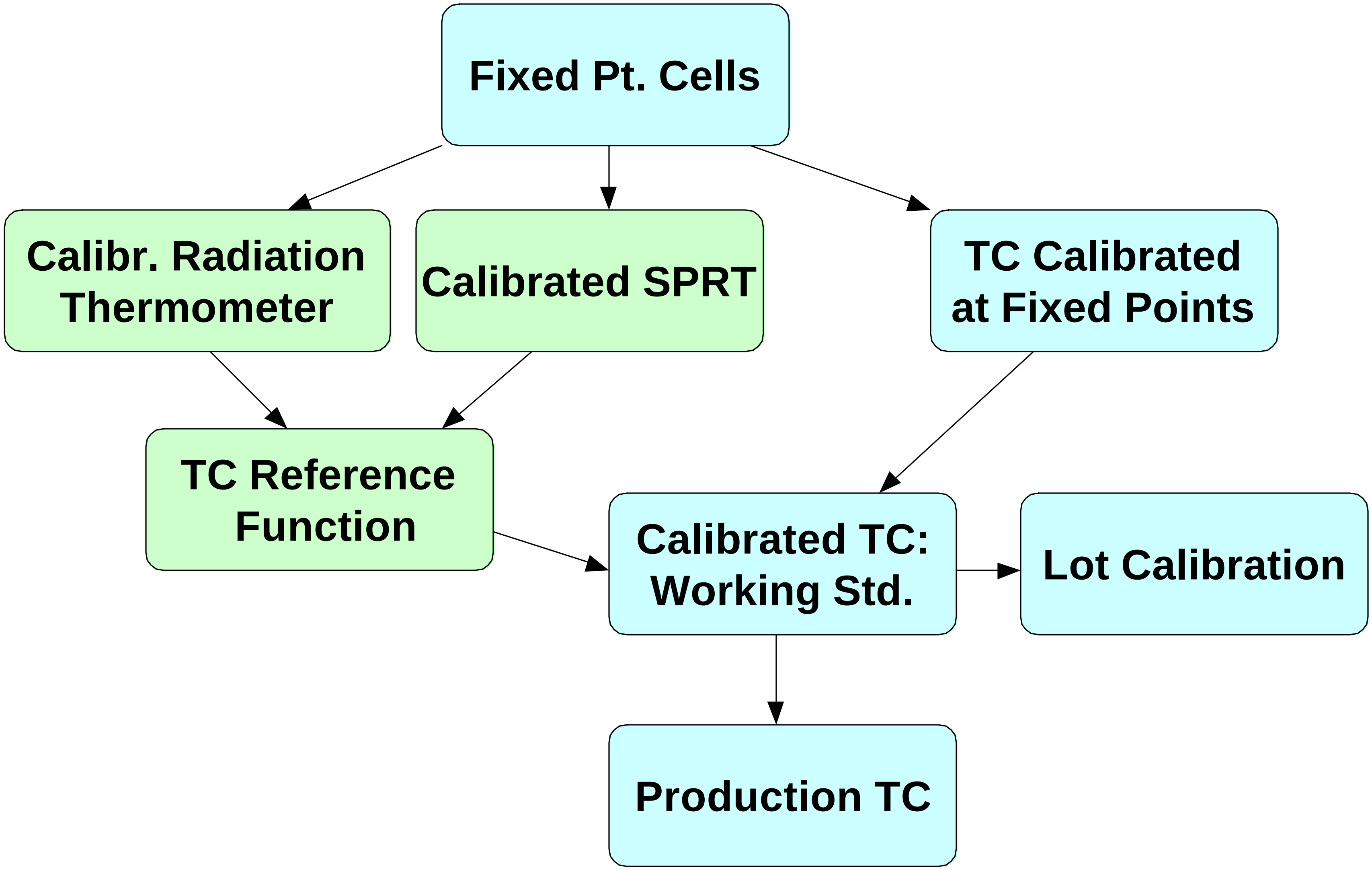
Use type R or S below 1300 °C, type B above 1300 °C.

Platinel: High Seebeck coefficient with some of the stability of types B, R, and S.

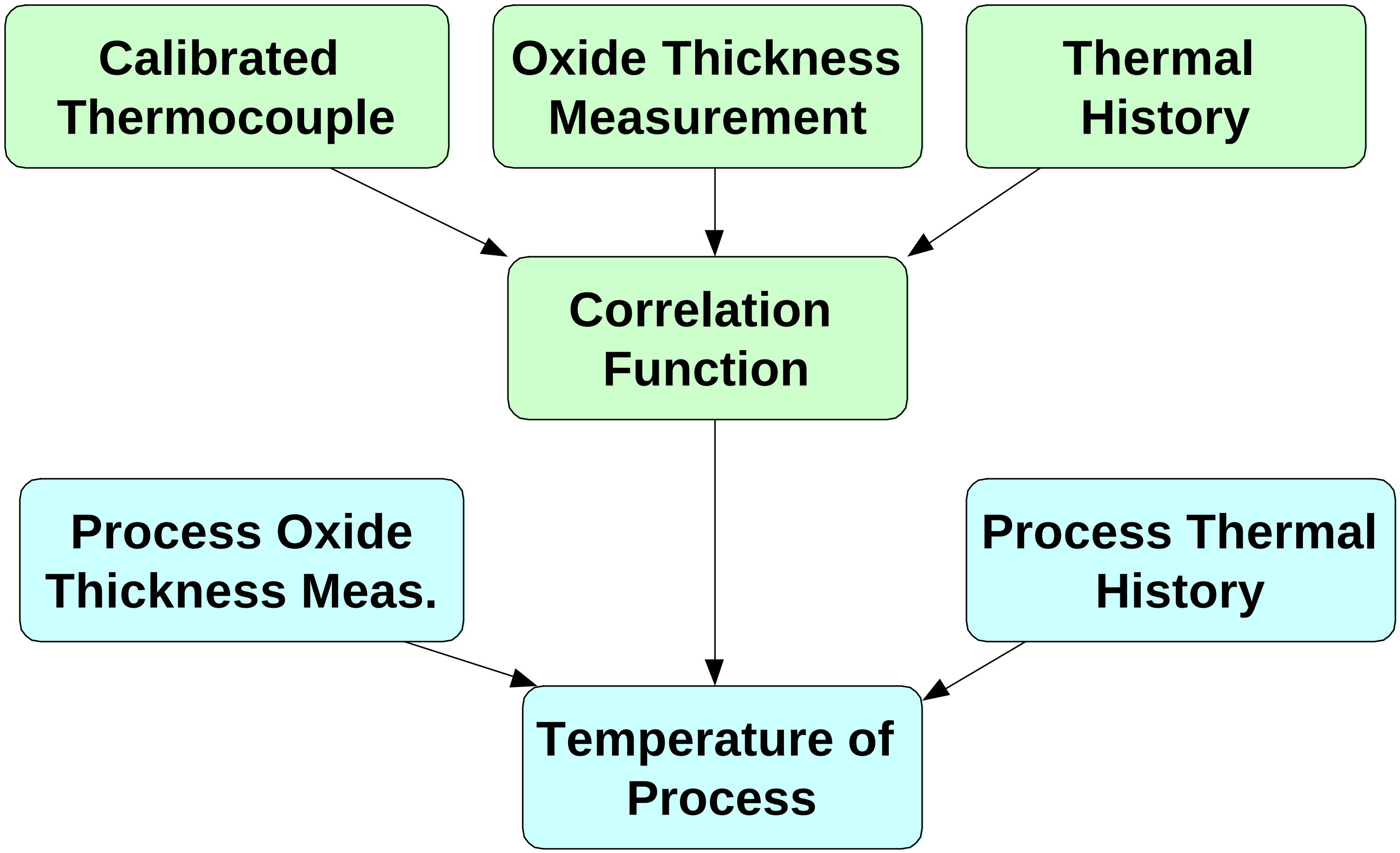
Au/Pt: The best accuracy from 0 °C to 1000 °C.

Pt/Pd: The best accuracy from 1000 °C to 1500 °C–not commercial

Traceability on the ITS-90



Traceability using Process Measurements



Traceability depends on the quality and ease of replication of original measurements

Thermocouple Construction Types

Bare wire with ceramic insulators

- the best performance for clean, high temp. environments

Soft-insulated wire

- polymer coatings excellent for use up to 200 °C
- woven ceramic sleeving–not well characterized

Mineral-insulated, metal-sheathed construction (MIMS):

- excellent for base-metal thermocouples at high temperatures
- excellent for unclean environments
- can be bent to shape

Bare wire with ceramic insulators, and outer metal sheath

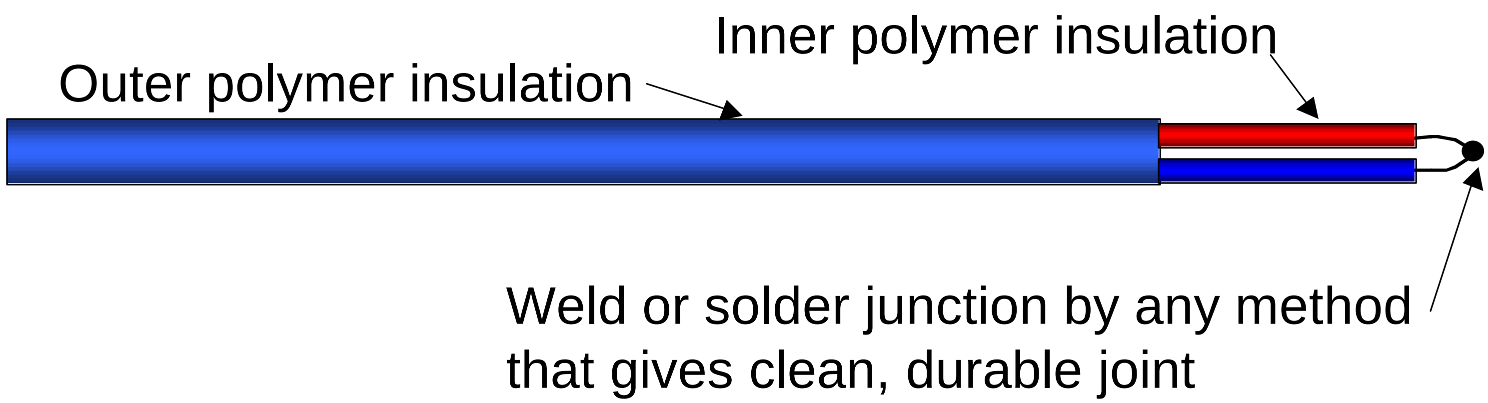
- not flexible
- better contamination resistance and less mechanical strain than MIMS construction for noble metal thermocouples

Thin-film thermocouples: discussed by K. Kreider in this Workshop

Bare Wire with Ceramic Insulators

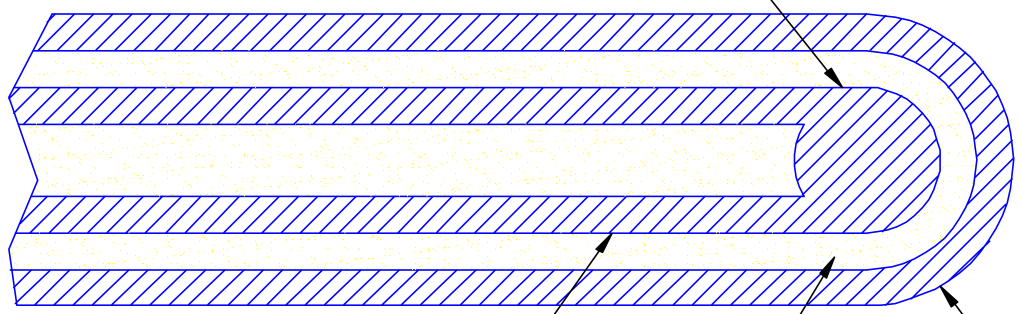
- For noble-metal alloys, use high-purity alumina (99% for typical uses, 99.7% for highest accuracy and stability).
- If old insulators are used, avoid cross contamination. e.g.: Pt wire into a bore that held Pt-Rh, or other base metals into bore that held iron
- Above 1300 °C, alumina insulator itself is a source of impurities.
- Use single, unbroken lengths of ceramic, to prevent contamination and loss of volatile alloy components
- Pt-Rh alloys annealed pre or post assembly for best performance

Soft-Insulated Thermocouples



- Choose polymer insulation based on upper temperature limit
- Woven ceramics are popular in semiconductor applications
  - Always bake out binders to avoid contamination
  - Contamination of thermocouples by ceramic has not been studied well
  - Use single lengths of alumina in high-gradient zone, if possible
  - Contamination is more of an issue with diffusion furnaces than RTP applications (much less time at temperature)

Mineral-Insulated, Metal-Sheathed (MIMS) Thermocouples



- At high temperatures, choice of sheath material is critical
  - for types K and N, sheath material dominates performance
  - for noble metal, Pt-Rh sheaths preferred. Large problems with contamination/strain with non-Pt-Rh sheaths
- MIMS thermocouples are available in small diameters (0.25 mm)
- Sheath protects thermoelements from contamination

Thermocouple Color Codes

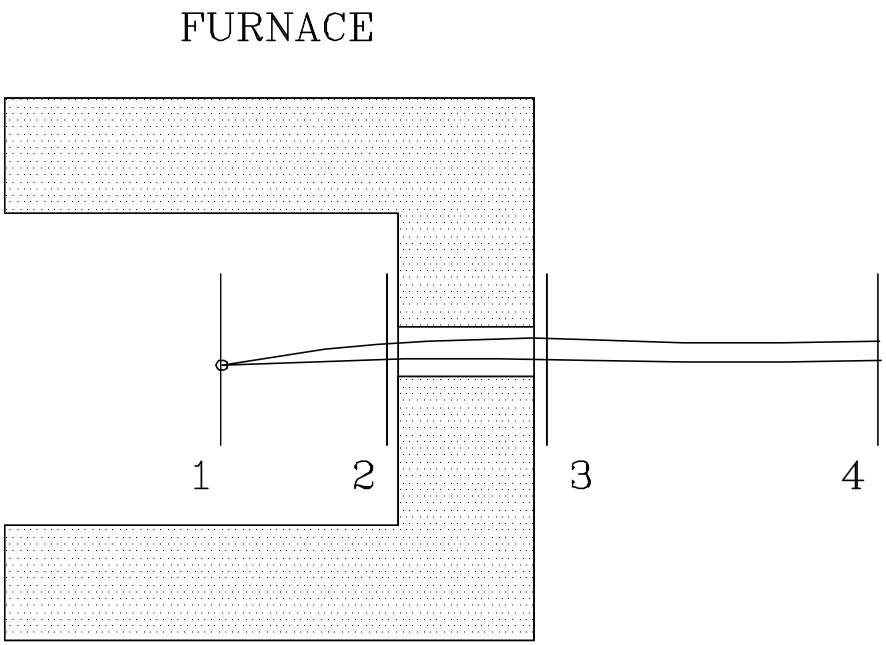
TC Type	IEC Positive Cond., Extension Sheath	ASTM Extension Sheath	ASTM Positive Conductor
B	—	Gray	Gray
E	Violet	Purple	Purple
J	Black	Black	White
K	Green	Yellow	Yellow
N		Orange	Orange
R,S	Orange	Green	Black
T	Brown	Blue	Blue

IEC: Negative Conductor is **White** for all Types

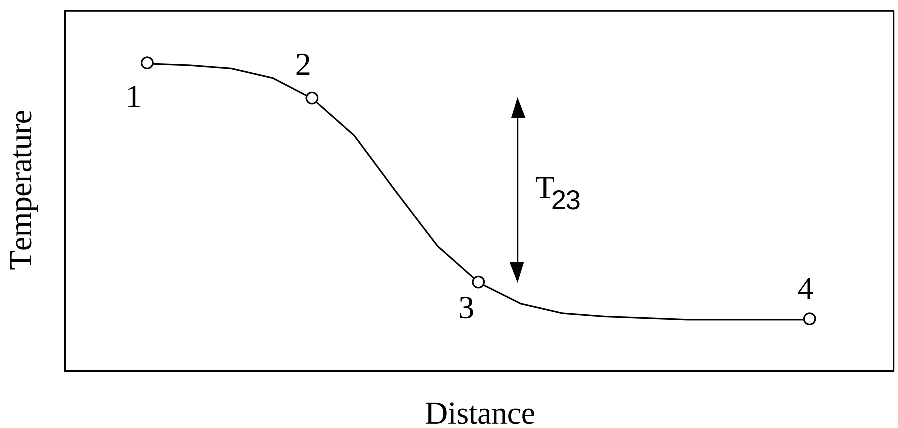
ASTM:

- Negative Conductor is **Red** for all Types
- For base metal types, duplex insulated thermocouple wire has identical color codes, but with brown overall insulation.

A Key to Understanding Thermocouples...



Thermocouples generate signal primarily in regions of strong thermal gradients. (Region 2-3)



$E_{12} = S_{12}\,(T_1 - T_2)$ Small
$E_{23} = S_{23}\,(T_2 - T_3)$ Large
$E_{34} = S_{34}\,(T_3 - T_4)$ Small

S_{12} = average Seebeck coefficient between points 1 and 2

Fundamental Laws of Thermoelectric Circuits

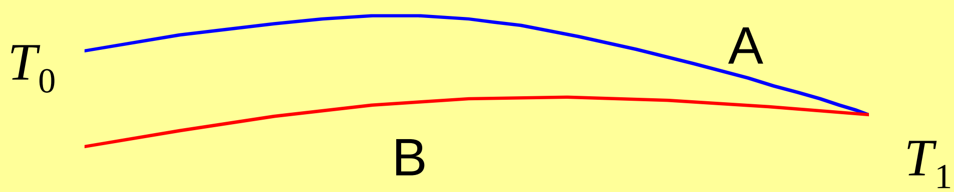
I. EMF OF HOMOGENEOUS WIRE

A homogeneous piece of wire (uniform chemical composition, uniform metallurgical state) will generate no emf if the two ends are at the same temperature, regardless of the temperature between the endpoints.

Example: hook up a copper wire to the inputs of a DVM and immerse the loop into liquid nitrogen. The measured emf will be very small.

A homogeneous piece of wire passing through a thermal gradient will develop an emf, $E(T_0,T_1)$, between its two ends. The emf depends only on the temperature of the end points; cross section is irrelevant.

A pair of wires:



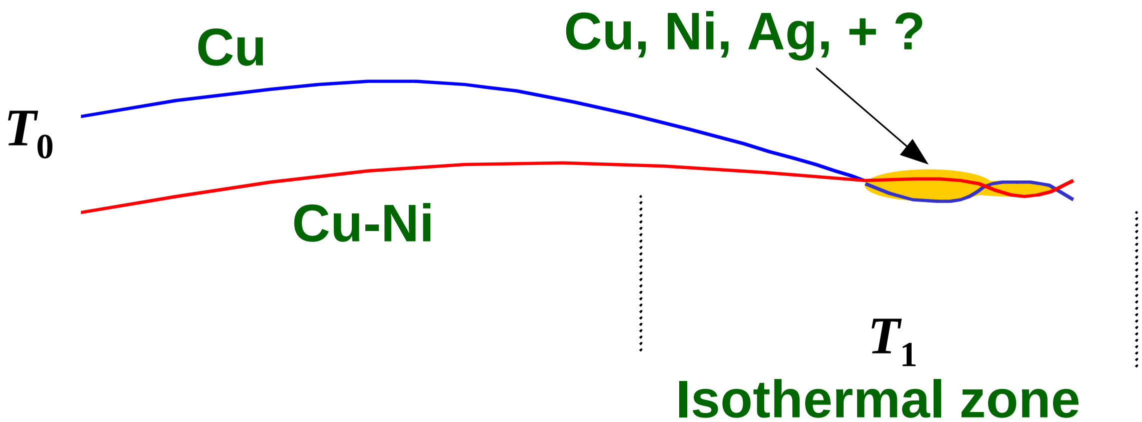
E = emf generated by the loop of wire

$$= E_A(T_0,T_1) + E_B(T_1,T_0) = E_A(T_0,T_1) - E_B(T_0,T_1)$$

II. EMF OF INHOMOGENEOUS WIRE

An inhomogeneous piece of wire will generate no emf if the whole piece is at a uniform temperature.

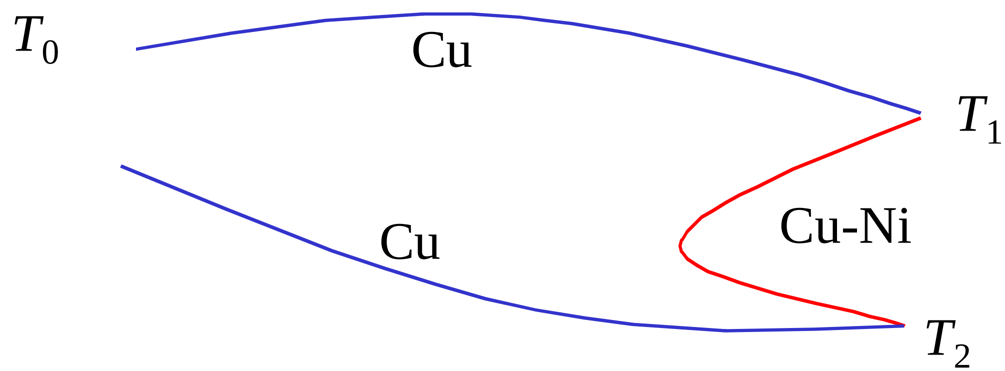
Example: A thermocouple junction is made by brazing, with a silver alloy, a copper and a constantan wire. The junction is placed in an isothermal zone of a furnace. The emf generated by the thermocouple is independent of the composition at the junction because the junction is at a uniform temperature.



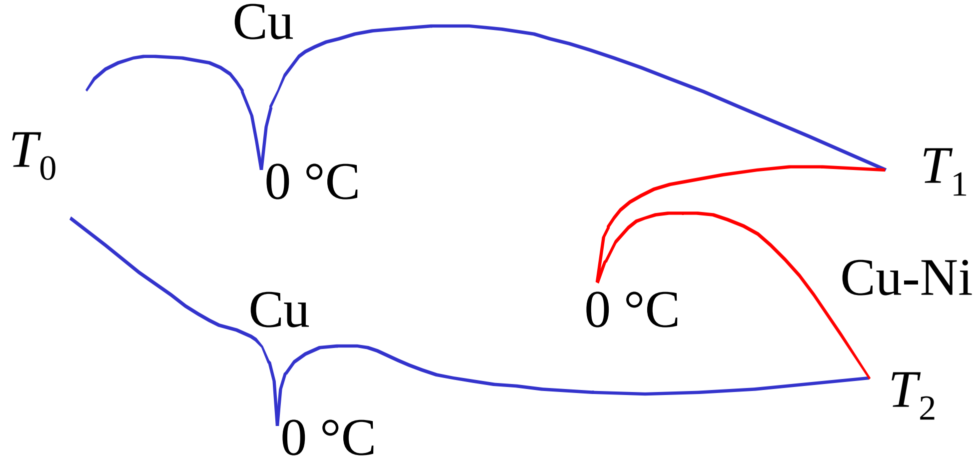
III. EQUIVALENT THERMOCOUPLE CIRCUITS

For thermocouple circuits with reference junctions at a temperature different from 0 °C, an equivalent circuit can be drawn with 0 °C reference junctions. Example: a differential thermocouple pair made of copper/constantan.

Actual circuit:

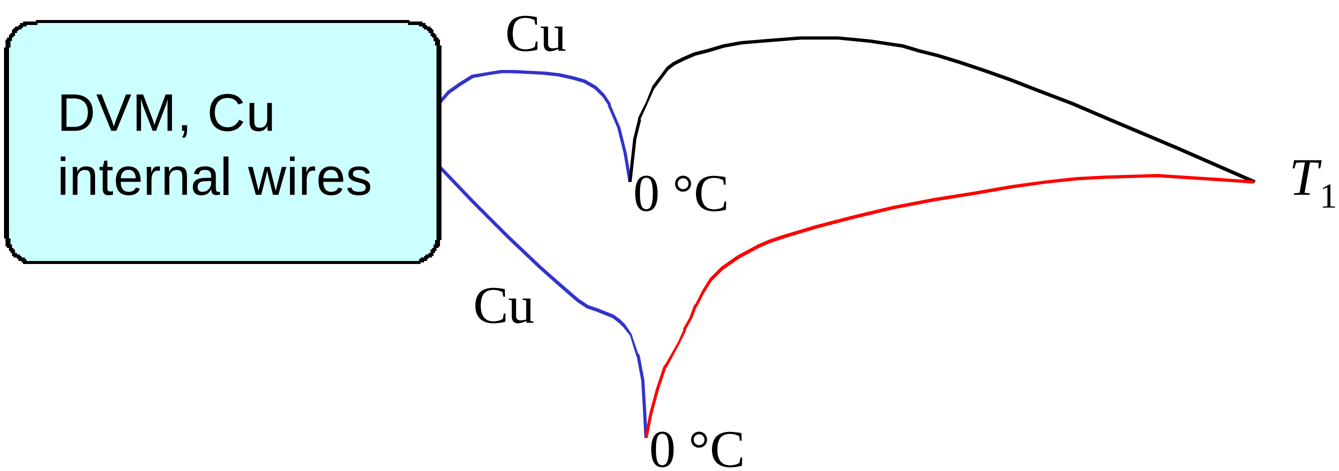


Equivalent circuit:



Typical thermocouple circuit

Reference junctions maintained at 0 °C by
- immersion into ice bath, made from water/ice slurry
- immersion into thermoelectrically-cooled, sealed ice bath



Reference
junctions at 0 °C

If Reference junctions are not at 0 °C, compensation of emf signal is necessary, by appropriate addition of voltage

Thermocouple Electrical Characteristics

Electrical characteristics

- low resistance (<100 Ω)
- low DC voltage (<40 mV)

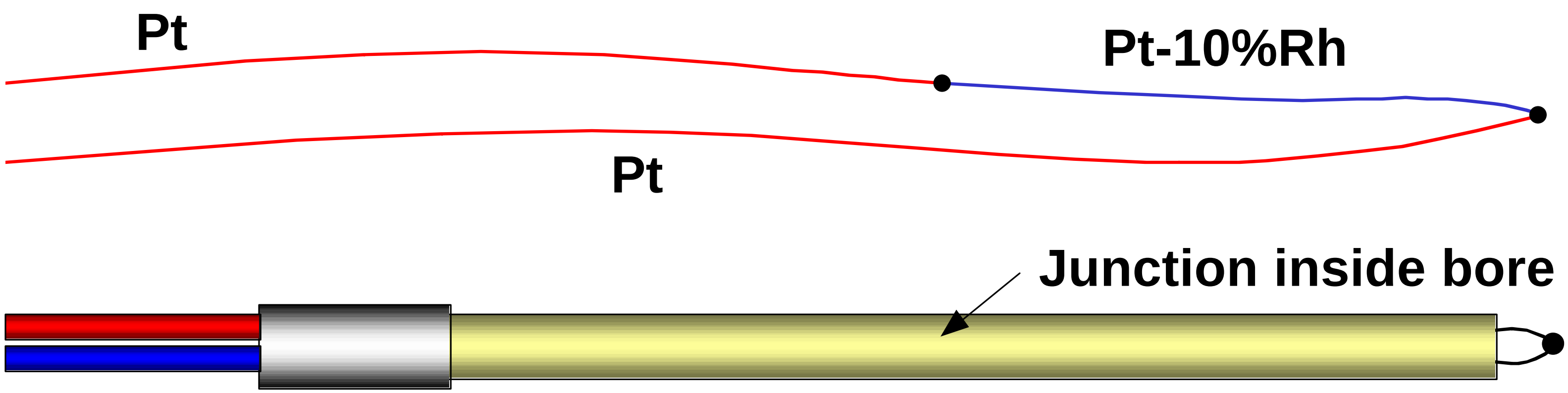
Sources of DC noise

- Extraneous thermal emf (voltmeters, scanner relays, wiring)
- Offset voltages (voltmeters, analog-to-digital converters)
- At high temperatures, electrical leakage through poor insulators

Sources of AC noise

- Magnetic pickup. Use twisted pair leadwire, keep thermoelements close.

Differential Thermocouple Pair



A differential thermocouple pair measures the temperature difference between two points directly

Advantages:

- Very accurate
- Reference junctions must be isothermal, but at any temperature

Disadvantages:

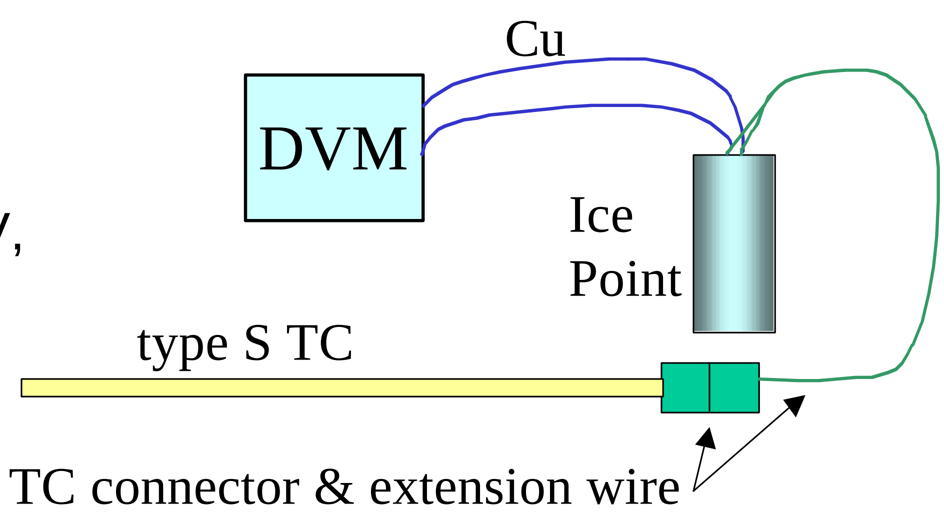
- No measure of absolute temperature at either junction
- Not common commercially

Superior measure of furnace uniformity compared to individual thermocouples in multiple zones

Extension Wires and Cables for Thermocouples

- Extension wires are fabricated from Cu-Ni alloys that are designed to mimic emf response of the standard thermocouple types
- If both ends of the extension wire are within 1 °C, the error in using extension wire is generally negligible
- If there is a large temperature difference between the ends, the error can be dominant
- Emf readings can be corrected, if the temperature difference between the ends remains constant

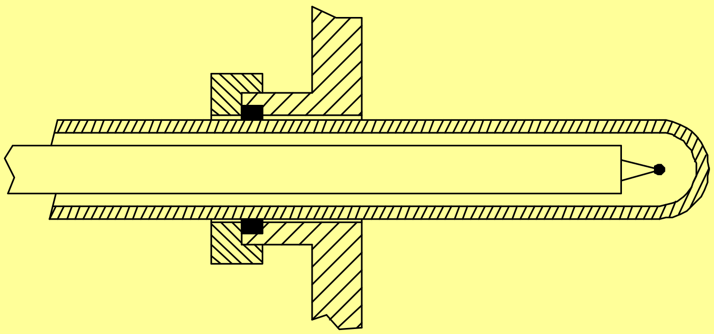
Example: type S extension wire from 0 °C to 23 °C introduces an error of ≈15 μV, equivalent to 1.4 °C for measurement of 900 °C



Hermetic Feedthroughs for Thermocouples

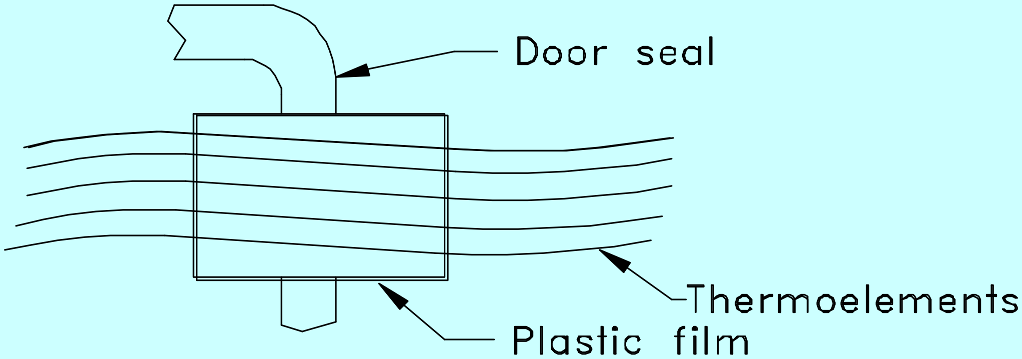
Compression fitting on outer sheath

- Simple
- Poor thermal contact
- Straight insertion only



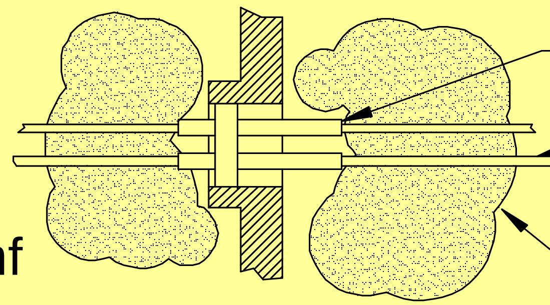
Thermoelements laminated between adhesive film

- Simple
- Not truly hermetic
- Wires may kink at door



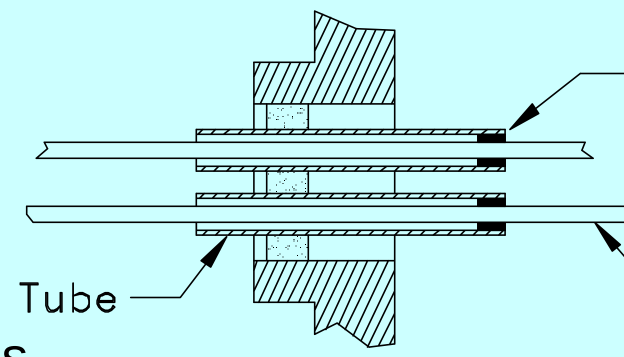
Hermetic Feedthroughs for Thermocouples

Thermoelements attached to hermetic feedthrough



- No leaks
- Possible extraneous emf
- Difficult to change TC

Thermoelements pass through feedthrough



- No leaks
- Difficult to manufacture
- Solution for unusual TCs

Limitations on Thermocouple Performance

- **Intrinsic variations in alloy composition (small, unavoidable)**
- **Chemical contamination (potentially very large)**
- **Physical strain (moderate: see Bentley in Resources)**
- **Preferential oxidation, volatilization (potentially large)**
- **Hysteresis in structural phase changes (small to moderate, worst for type E and K)**
- **Extension wires, thermocouple connectors, feedthroughs (potentially large, but avoidable)**

Typical values for each effect, *T* » 900 ° C

	Base	Noble
small	1 °C	0.1 °C
moderate	3 °C	1 °C
large	³10 °C	³ 3 °C

Intrinsic Variations in Thermocouple Homogeneity

There are unavoidable variations in thermoelectric properties along the length of a wire caused by:

- compositional variations in the wire alloy
- variations in the annealing state

In the best circumstances, the fractional uncertainty $\Delta T/T$ of measuring a temperature interval $T_1 - T_2$ is very approximately:

Base metal	10^{-3}
Pt-Rh alloy	10^{-4}
Au/Pt or the best Pt/Pd	10^{-5}

This level of performance requires careful manufacture and use

Chemical Contamination

- Pure elements are much more sensitive to impurities than alloys
- In semiconductor processing, absence of oxygen may lead to reduction of oxides and increased levels of free impurities such as Si

Examples of the sensitivity of Pt to impurities

Element	$\Delta E/\mu V$ at 1200 °C per 10^{-6} mass impurity
Cu	0.12
Fe	2.30
Cr	4.04
Mn	0.32
Si	1.17

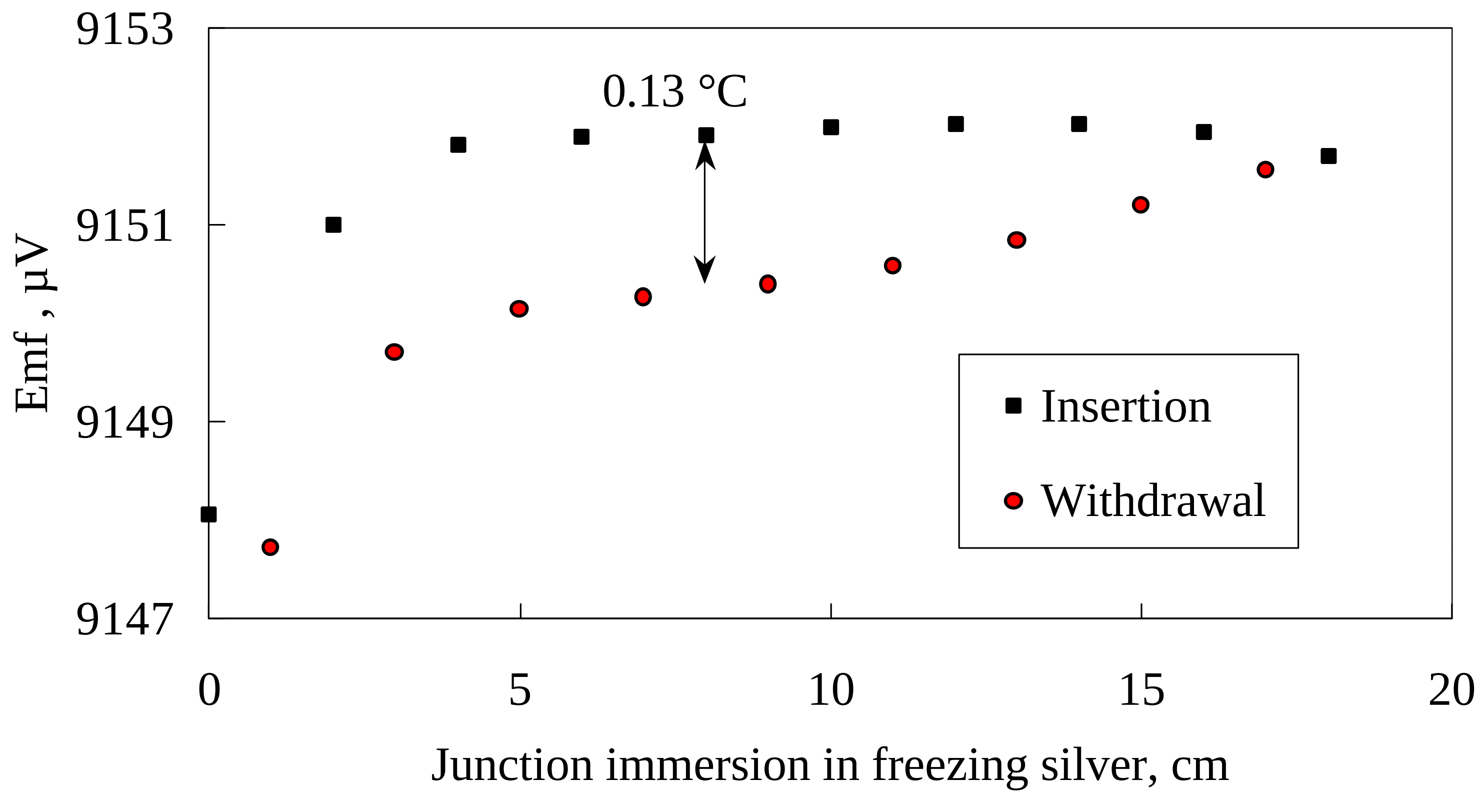
(at 1200 °C, 1 μV equivalent to 0.08 °C for type S)

Cochrane, *Temperature*, Vol. 3, p. 1619 (ISA, 1973)

Preferential Oxidation in Pt-Rh Alloy Thermocouples

Example: emf of a type S TC at Ag freezing point (961.78 °C) is altered as Rh changes oxidation state during test.

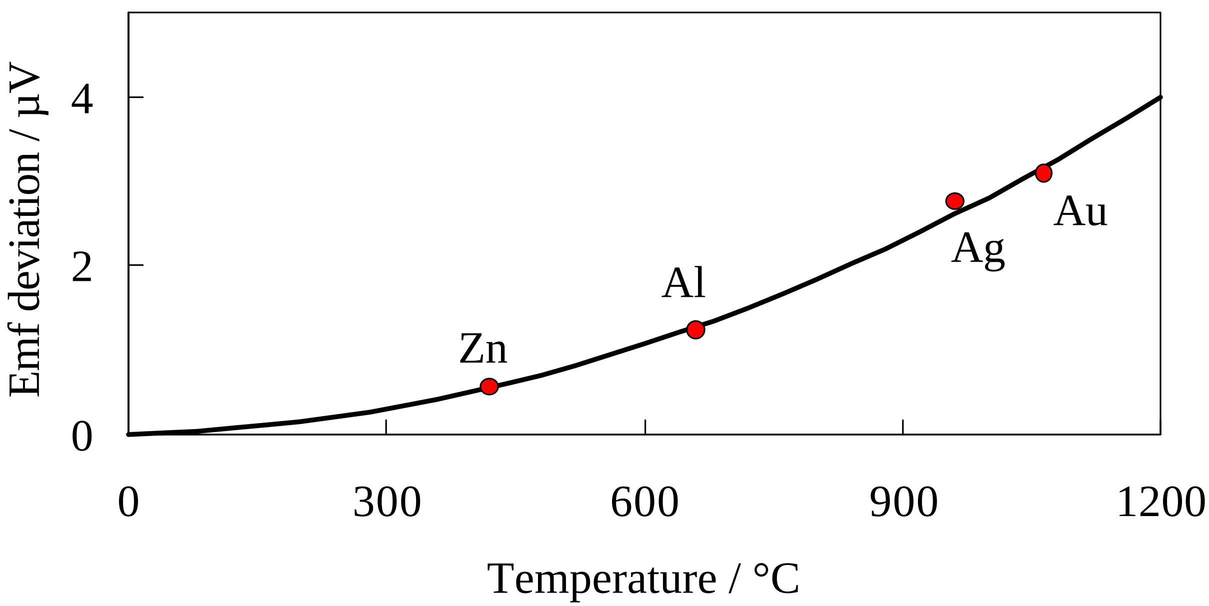
- Effect is 3X larger for some cases of rapid temperature change.
- Effect is reversible, unless oxide is volatile and sublimates



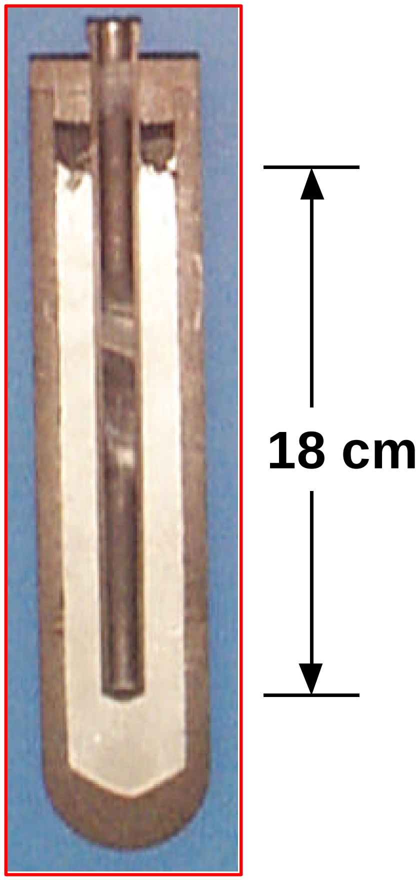
Calibrations in Fixed-point Cells

- Fixed point uncertainty <2 mK for Ag and below
- Test uncertainty dominated by stability of test thermocouple

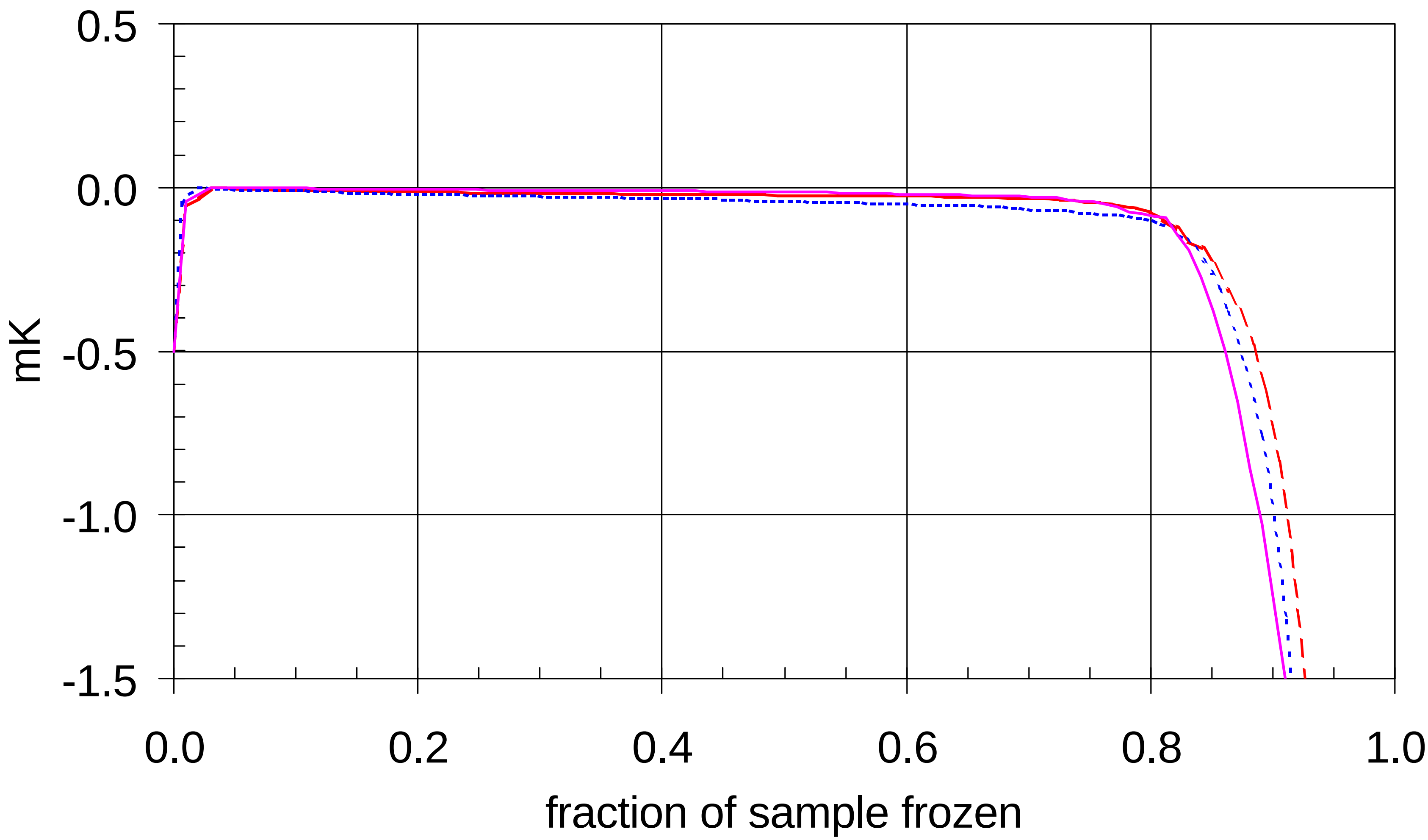
Type S TC Fixed-Point Calibration



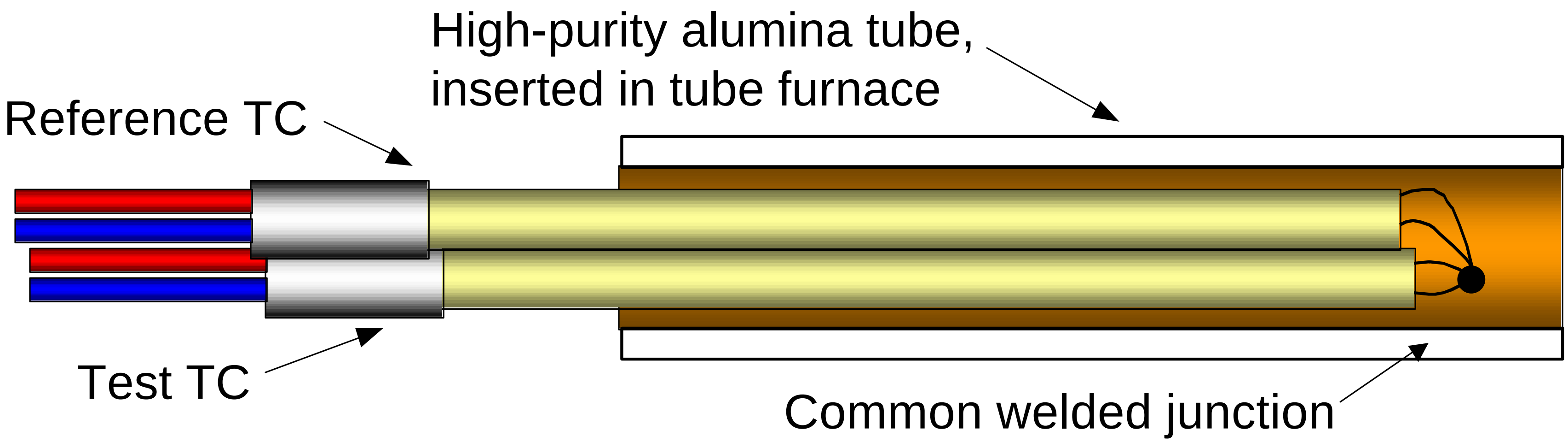
Cross section of a metal freezing-point cell



Example of a Fixed-point: Freezing Curves of an Indium Fixed-point Cell



Comparison Calibrations in Furnaces



- TCs measured simultaneously, to cancel temperature drift
- Measuring junctions at center of furnace to minimize gradients
- Reference TC calibrated at fixed points

NIST Uncertainties in °C

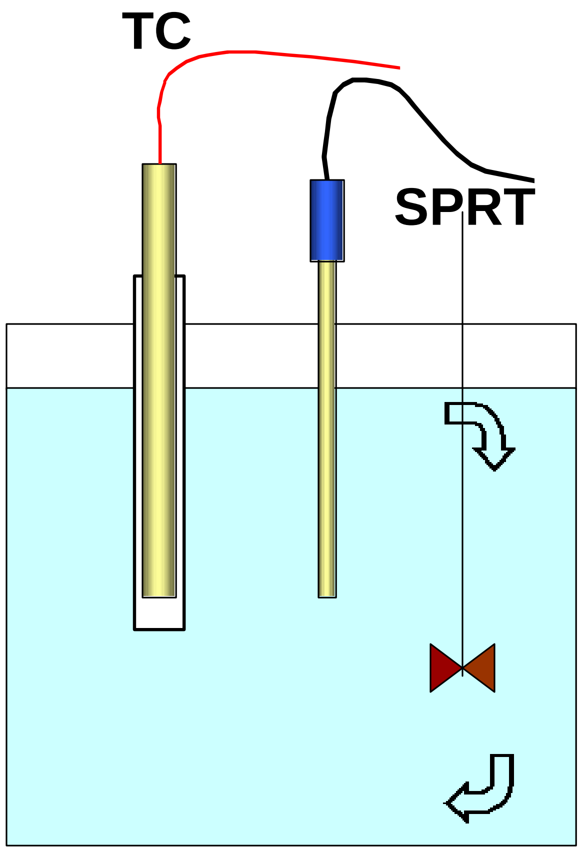
	Base TCs	**Noble TCs**
0 °C	0.1	0.1
400 °C	0.4	0.2
900 °C	1.0	0.3

Comparison Calibrations in Stirred-liquid Baths

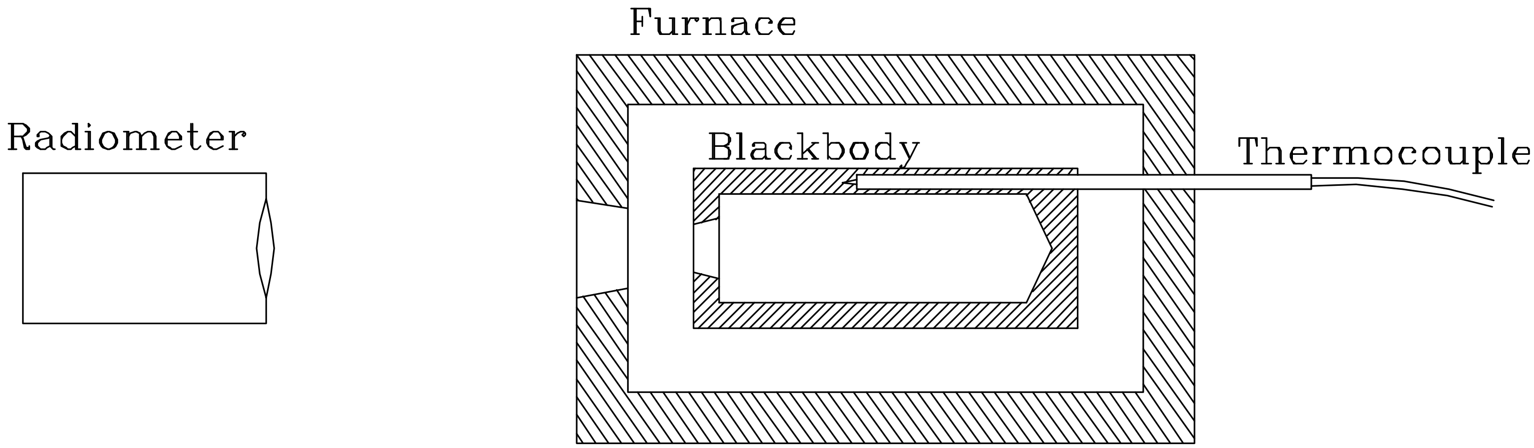
- SPRT uncertainty <1 mK (negligible)
- Bath stability, gradients <20 mK (generally negligible)
- Test uncertainty dominated by stability of test thermocouple

Operating range:

ethanol	–90 °C to 5 °C
water	0.5 °C to 95 °C
oil	95 °C to 275 °C
salt	275 °C to 550 °C



Calibration of Thermocouples above 961.78 °C



Temperatures on the ITS-90 above 961.78 °C defined by radiation thermometry

1. Calibrate radiometer on the ITS-90 at a single fixed-point temperature: silver, gold, or copper.
2. Place thermocouples in or near the blackbody.
3. Measure the emf of the thermocouple while simultaneously measuring the blackbody temperature radiometrically.

Difficult and expensive:

Often only done in determination of reference function

Calibration Uncertainty Components

Temperature reference

- reference calibration uncertainty
- reference stability
- readout uncertainty
- reference junction temperature

Test thermocouple

- test thermocouple stability and homogeneity
- readout uncertainty, including effects of extraneous emf

Thermal equilibrium of test and reference

- Comparison bath/furnace stability
- Comparison bath/furnace uniformity

Method for Evaluation of Uncertainty

ISO Guide to the Expression of Uncertainty in Measurement

1. Evaluate uncertainty components by statistical analysis of data: Type A

   or

   Evaluate by other methods (calculation, calibration cert., etc.): Type B

2. Express all uncertainties at the one standard deviation level.
   Standard uncertainty = u.

3. Combine all uncertainties using the Law of Propagation of Errors, equivalent to root-sum-of-squares (RSS) in simple cases.

4. Expanded uncertainty = $U = ku_c$, where k=coverage factor, often 2.
   If uncertainties are not normally distributed, establish confidence limits for stated k value.

Important point: RSS strongly weights only the few largest uncertainty components. Emphasize careful evaluation of these few components!

Uncertainty Budget for Type S TCs at Fixed Points

Uncertainties in µV	Au (≈1064 °C)	Ag (≈962 °C)	Sb (≈630 °C)	Zn (≈420 °C)
Type B				
Emf Measuring System	0.05	0.04	0.03	0.02
Temperature of Liquidus Point	0.03	0.02	0.08	0.01
Change in Liquidus Point	0.10	0.09	0.06	0.06
Thermocouple Sheath Losses	0.07	0.07	0.06	0.06
Reference Junction Temperature	0.02	0.02	0.02	0.02
Total Type B	**0.14**	**0.12**	**0.12**	**0.09**
Type A				
Uncert. of Check-Standard	0.43	0.35	0.38	0.27
Uncert. of Quadratic Function	0.20	0.20	0.20	0.20
Total Type A	**0.47**	**0.40**	**0.43**	**0.34**
Expanded Uncertainty, $U=2u_c$	**1.0**	**0.8**	**0.9**	**0.7**

Care and Feeding of Thermocouples

Noble Metal Thermocouples:

- Use at the same immersion at which the calibration was performed.
- Protect from contamination by the furnace environment, using single lengths of alumina insulator when possible
- Protect from mechanical strain and kinks

Base Metal Thermocouples:

- Monitor drifts in base metal thermocouples by *in situ* tests
- Protect from contamination using alumina or silica tubes, or use mineral-insulated-metal-sheathed thermocouple wires.
- For each temperature environment to be measured, a new thermocouple should be made, and it should always be used at the same immersion.
- Obey the ASTM upper temperature limits for bare wire thermocouples.

The Difficulty of Recalibrating Used Thermocouples

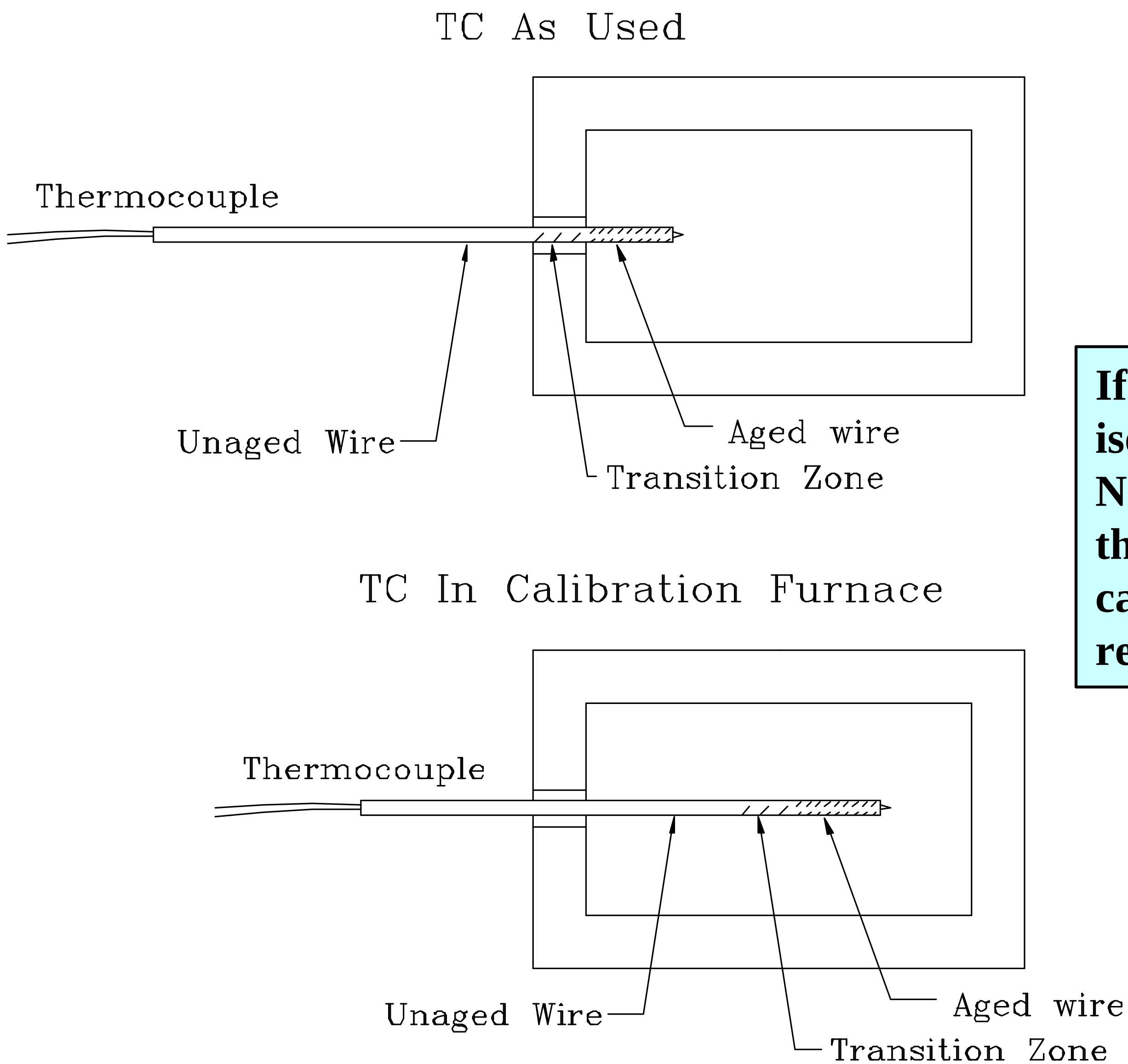
- With use at elevated temperatures (>200 °C to 400 °C), thermocouples become inhomogeneous.

- Calibration of a used thermocouple in a different apparatus often will produce **meaningless** results! Often, only *in situ* recalibration is meaningful.

- Noble metal thermocouples can be partially restored to a homogeneous state by annealing electrically or in a furnace. Base metal thermocouples are generally not reannealed.

- Any recalibration of a used thermocouple should include a check of the thermocouple homogeneity. (Example: test at different immersions.)

Example of a Misleading Calibration

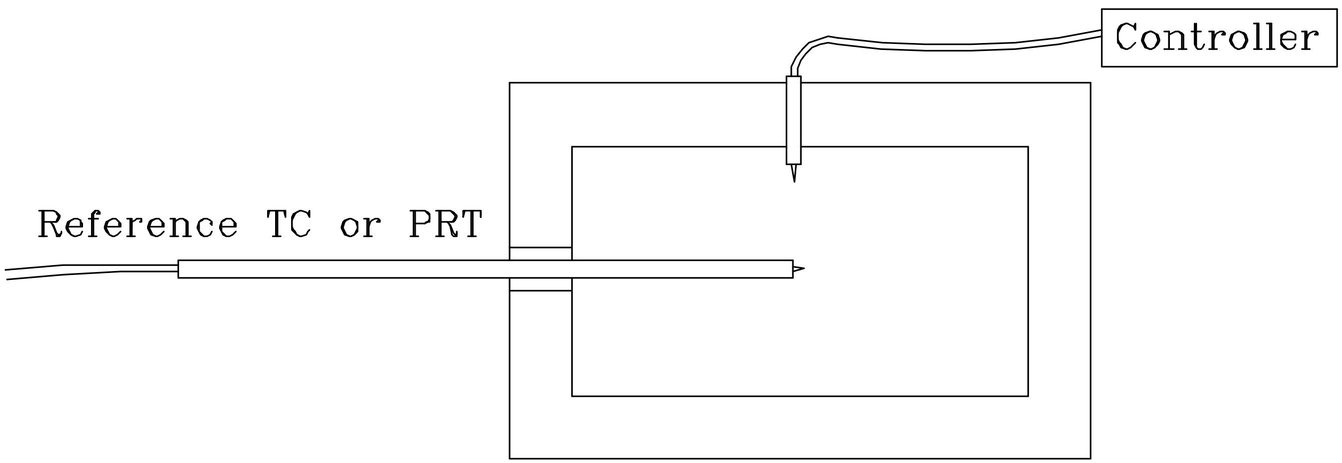


If the furnace is isothermal, there will be NO difference between the original TC calibration and the recalibration.

Alternatives to Recalibration of Used Thermocouples

Option 1. Recalibrate thermocouples *in situ*.

Example: a reference thermocouple is inserted into a furnace with a control thermocouple. The control thermocouple may be recalibrated without removal.



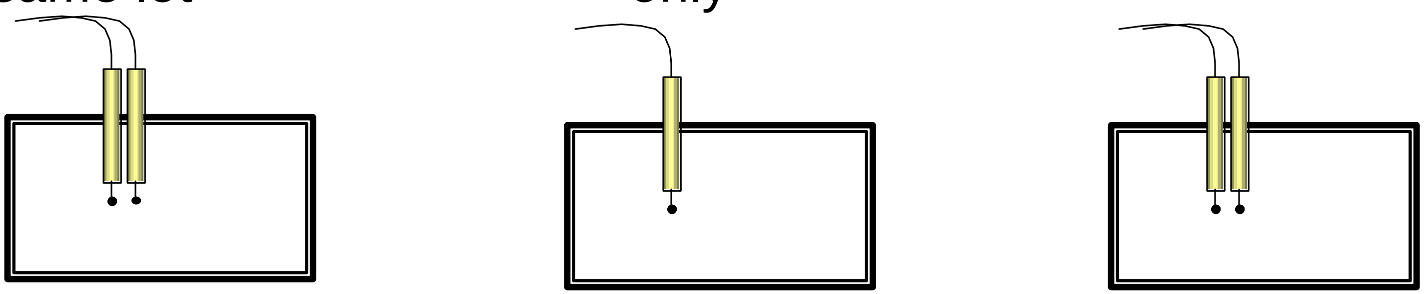
Alternatives to Recalibration of Used Thermocouples

Option 2. Periodic Replacement. Determine a typical drift rate of thermocouples in an application and replace thermocouples periodically.

Drift rate may be determined by:

- Finding similar cases documented in the literature,
- *In situ* calibrations,
- *In situ* comparison measurements between two thermocouples of the same lot, one of which is used and one of which is new.

2 TCs from same lot | Process TC only | 2 TCs again



day 1 | day 2 through 119 | day 120

Resources

Books

- *Traceable Temperatures*, J. V. Nicholas and D. R. White (John Wiley, 1994)
- *The Theory and Practice of Thermoelectric Thermometry*, Vol. 3 of the *Handbook of Temperature Measurement*, R. Bentley (Springer, 1998)
- *Manual on the Use of Thermocouples*, ASTM, MNL-12 (ASTM, 1993)

Links

- ASTM standards and tables: www.astm.org
- NIST Thermometry Group : www.cstl.nist.gov/div836/836.05
- General reference on thermometry: www.temperatures.com

Training

NIST Precision Thermometry Workshop, every March and October. Contact Andrea Swiger at andrea.swiger@nist.gov.